



## Stress induced martensite at the crack tip in NiTi alloys during fatigue loading

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**ABSTRACT.** Crack tip stress-induced phase transformation mechanisms in nickel-titanium alloys (NiTi) were analyzed by Digital Image Correlation (DIC), under fatigue loads. In particular, Single Edge Crack (SEC) specimens, obtained from a commercial pseudoelastic NiTi sheet, and an *ad-hoc* experimental setup were used, for direct measurements of the near crack tip displacement field by the DIC technique. Furthermore, a fitting procedure was developed to calculate the mode I Stress Intensity Factor (SIF), starting from the measured displacement field. Finally, cyclic tensile tests were performed at different operating temperature, in the range 298-338 K, and the evolution of the SIF was studied, which revealed a marked temperature dependence.

**KEYWORDS.** Shape Memory alloys; Fracture; Digital Image Correlation; Stress Intensity Factor.

### INTRODUCTION

In last decades, shape memory alloys (SMAs), and in particular the Nickel Titanium-based ones (NiTi), received big attention from scientific and engineering communities thanks to their unique characteristics, namely shape memory effect (SME) and superelastic effect (SE) [1]. In particular, these properties allow large recoverable strains or large induced internal forces due to a reversible solid-state phase transformation between austenite and martensite; this transformation can be activated by a temperature variation (TIM, thermally induced martensite) or by the application of external forces (SIM, stress-induced martensite). Due to these interesting features, as well as to their good mechanical performances and biocompatibility, NiTi alloys have seen growing use in many branches of engineering and medicine [2-3]. As a direct consequence of this interest, many studies were carried out to better investigate on the thermo-mechanical behavior of SMAs, in terms of both SME and SE [1].

Their mechanical properties were investigated using tensile (e.g. [4-5]) and fatigue testing (e.g. [6-11]), however, many aspects are still unknown, especially because the hysteretic stress and/or thermally induced phase transformations significantly affect the damage mechanisms occurring under fatigue loadings, i.e. the crack formation and propagation mechanisms. From a materials science point of view, an accurate knowledge of these topics is essential for predicting the functional and structural life of damaged structures as well as their failure modes, with the aim to improve the overall performances of NiTi-based components or structures. The fracture behavior of austenitic NiTi alloys strongly depends on the SIM transformation which occurs in the crack tip region, as a consequence of the high values of local stresses. As a direct consequence of the marked non-linear and hysteretic behavior, classic elastic and/or elastic-plastic theories cannot be directly applied to SMAs.

The constitutive laws implemented in commercial finite element software are based on phenomenological approaches and on theory of plasticity, i.e. they use plasticity-like concepts to describe the effects of phase transformation mechanisms on

the macroscopic response of NiTi alloys [12]. Even though these numerical methods represent useful design tools to simulate the macroscopic response of simple or complex SMA based systems, special care should be taken when they are used to study the local effects in the proximity of high stress concentration regions. In particular, it was demonstrated that the high values of local stresses arising in the crack tip region of NiTi alloys cause stress-induced phase transition mechanisms [13-30], which significantly affect the crack tip stress distribution and, consequently, the crack evolution under both static and fatigue loading conditions. Despite the increasing number of research activities on fracture and fatigue of NiTi alloys in recent years, much effort should be devoted for an effective understanding of the role of the phase transformations in the crack formation and propagation mechanisms and in the stress state generated at the crack tip.

Within this context, the development and application of full field techniques to analyze the local transformation mechanisms near geometrical discontinuities and, in particular, in the crack tip region, represent a highly challenging scientific goal. For this purpose, synchrotron X-ray micro-diffraction (XRD) [13-15], infrared thermographic (IR) [16] and Digital Image Correlation (DIC) [18, 19] techniques were recently applied, to better understand the mechanisms of phase transformation in the notch and/or crack tip proximity. In particular, a pseudoelastic NiTi alloy for medical applications were analyzed in [13] by using miniature compact-tension (CT) specimens, which were directly obtained from thin-walled tubes, similar to those used for manufacturing self-expanding stents. XRD microdiffraction investigations of fatigue pre-cracked specimens revealed that the crack tip local strain are due to both B2 to B19' transformation and to the subsequent loading of the martensitic phase. Strain and texture evolution near the crack tip of a martensitic NiTi alloy were analyzed in [14], by synchrotron X-ray experiments, after fatigue crack propagation in a compact-tension (CT) specimen; it was found that texture evolution is mainly due to detwinning, the main deformation mechanism in martensitic NiTi alloys. Both martensitic and austenitic alloys were analyzed in [15], by using miniaturized CT specimens [19] after fatigue crack propagation, which revealed the presence of detwinned martensite at the crack tip of both martensitic and austenitic specimens.

An austenitic NiTi alloy was analyzed in [17] by DIC analysis of thin edge cracked specimen, which allowed direct measurement of the crack tip strain field related to stress-induced transformation. These experimental investigations, together with other several recent numerical [20-22] and analytical [23-30] studies, provide very useful information about the occurrence of crack tip transition mechanisms in NiTi alloys subjected to static and/or monotonic loads, and the effect of the operating temperature on the stress intensity factor (SIF) and on the size of the transformation region was also numerically investigated in [30]. However, the role of these mechanisms on the fracture properties on NiTi alloys is not yet completely defined, i.e. no reliable methodologies were developed for an effective design against fracture. In addition, the evolution of microstructural changes at the crack tip occurring during fatigue loading, i.e. the thermal and mechanical hysteretic behavior, was not yet investigated. This topic is of major concern as the hysteretic nature of phase transitions is expected to play a significant role on the crack growth mechanisms.

In this work the stress intensity factor evolution in a NiTi pseudoelastic alloy was investigated by means of a full field experimental techniques. In particular, DIC method was used to analyze the displacement field in the crack tip region by which, with a proper fitting procedure based on the William's series expansion [31], the mode I stress-intensity factor (SIF) was evaluated. Different cyclic tests, a different operating temperatures, in the range 298-338 K, were performed and the effect of the temperature on the SIF was investigated.

## MATERIAL AND METHODS

A commercial nickel-titanium sheet (thickness  $t=0.5$  mm) with pseudoelastic properties at room temperature (Austenite finish temperature  $A_f=286.7$  K) was used in this investigation. Fig. 1a shows the isothermal ( $T=298$  K) stress strain ( $\sigma$ - $\epsilon$ ) response of the material obtained from a complete loading-unloading cycle up to a maximum deformation of about 6.2 %, corresponding to a complete stress-induced martensite transformation. The figure also reports the values of the main thermo-mechanical parameters of the alloy: transformation stresses ( $\sigma_s^{AM}$ ,  $\sigma_f^{AM}$ ,  $\sigma_s^{MA}$  and  $\sigma_f^{MA}$ ), transformation strain ( $\epsilon_L$ ), Young's moduli of austenite and martensite ( $E_A$  and  $E_M$ ) and Clausius-Clapeyron constants ( $C_A$  and  $C_M$ ).

Edge-notched tension specimens were cut from a commercial plate by electrical discharge machining (EDM) and a 0.1 mm EDM wire was used to machine the notch. The specimen surface has been properly treated in order to provide a suitable speckle pattern for using DIC, therefore, the specimens were not painted.

Specimens were fatigued by using a servo hydraulic testing machine (Instron 8500), at rate of 5 Hz, to initiate and grow a crack from the EDM notch tip at constant load amplitude and a load ratio,  $R = \sigma_{min} / \sigma_{max}$ , of roughly zero, in order to get a length ratio,  $a/W$ , close to 0.3 (see Fig. 2). Pre-cracked specimens were subsequently subjected to complete tensile loading-unloading cycles by using an electro-mechanical testing machine (MTS Criterion s42) equipped with 5 kN load cell; different tests were carried out at different operating temperature, in the range 298-338 K and with a maximum load  $P = 300$  N, corresponding to a maximum stress  $\sigma_{max} = P_{max} / Wt = 60$  MPa.

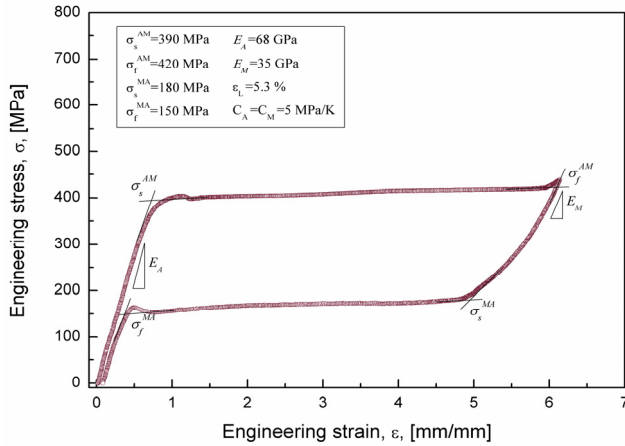


Figure 1: Loading-unloading isothermal stress-strain cycle (298 K)

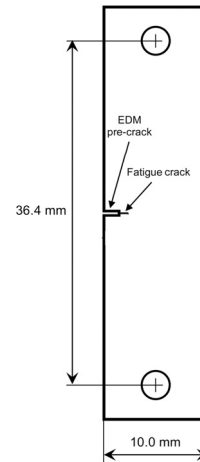


Figure 2: Single Edge crack specimen

A digital camera (Sony XCD- X910 model) with a resolution of 1280 by 960 pixels of  $4.65 \mu\text{m}$  was used to capture images throughout measurement tests. The focus of the images was performed using a Linos Photonics microscope objective with a 4x magnification and a numerical aperture of 0.1, which ensures, in conditions of correct illumination, a resolution of approximately  $2.5 \mu\text{m}$ , and therefore lower than the pixel size of the camera.

DIC was performed on images from each test carried at different operating temperature using a special tool available in the Matlab® software platform. The first image in the measurement cycle (at minimum load) was used as the reference image, and terms up to first order displacement gradients were used in all correlations. DIC was used to obtain full-field displacements for each image throughout the cycle and the correlations were performed using a subset size with a radius of 42 pixels and a spacing of 2 pixels between subset centers.

## RESULTS AND DISCUSSIONS

Cyclic tensile tests were performed on a single edge crack specimen, at different operating thermal conditions in order to understand the effect of the temperature on the stress induced transformation mechanisms at the crack tip. Three different values of temperature, in the range of 298-338 K, were adopted during the tests.

To determine the effective stress intensity factor the specimen actually experiences, which could be different than the theoretical one determined by the Linear Elastic Fracture Mechanic (LEFM) theory due to the marked non-linear behavior of NiTi alloys, a least squares regression was performed on the DIC measured displacements. The displacement field, at the crack tip, proposed by Williams [31], was used to fit the experimental data obtained from the tests.

As the displacement field is not unique unless rigid body motion is also specified, two in-plane rigid motion terms, rotation,  $A$ , and rigid translation perpendicular to the crack line,  $B$ , have to be specified. Furthermore, due to the low magnitude of the pictures, if the  $T$ -stress term is included, in order to improve the fitting of the analytical and experimental results, the asymptotic crack tip displacement equations become:

$$v = \frac{K_I}{\mu} \sqrt{\frac{r}{2\pi}} \sin\left(\frac{\theta}{2}\right) \left[ \frac{1}{2} (k+1) - \cos\left(\frac{\theta}{2}\right) \right] - \frac{1}{2\mu} \left( \frac{v}{1+v} \right) Tr \sin(\theta) + Ar \cos(\theta) + B \quad (1)$$

where

$K_I$  is the effective stress intensity factor,

$r$  is the distance from the crack tip,



$\theta$  is the angle from the crack line ahead of the tip,

$\nu$  is the Poisson's ratio,

$\mu$  is the shear modulus given by:

$$\mu = \frac{E}{2(1+\nu)} \quad (2)$$

and  $k$  is given by:

$$k = \frac{3-\nu}{1+\nu} \quad (3)$$

The vertical displacement filed, recorded at the maximum applied load ( $P=300$  N) for a specimen with  $a/W=0.32$ , is given in Fig. 3. The experimentally obtained and regressed displacements were plotted together to demonstrate the accuracy of the regression technique. In particular, blue contours represent the experimentally found displacements and the red contours represent the regressed displacement contours. As observed, the experimental and regressed displacement contours show good agreement.

For a given value of the Young's modulus, which was assumed to be constant and equal to that of austenitic untransformed structure ( $E_A=68$  GPa), the fitting procedure allows a direct estimation of the mode I stress-intensity factor ( $K_I$ ). However, it is important to underline that, due to the transformation mechanisms occurring in the crack tip region, the effective elastic properties changes, as consequence of the generation of the new phase and its variants reorientations, therefore further estimations should be carried out in this way to well characterize the real stress state involving the crack tip in a pseudoelastic NiTi alloy.

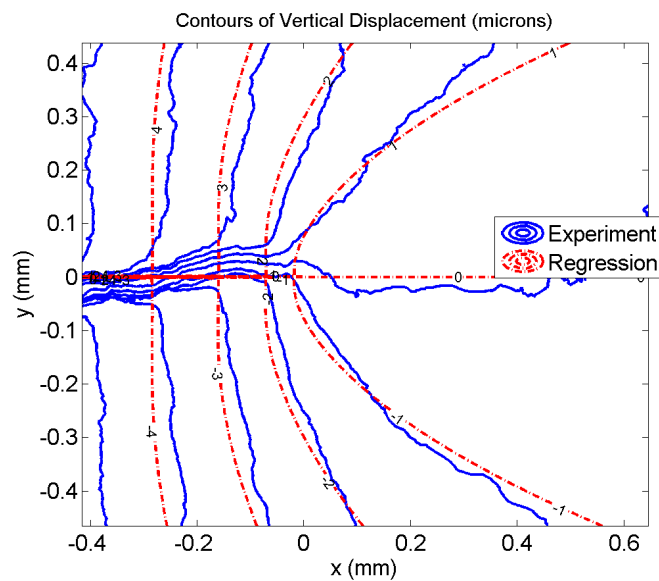


Figure 3: Comparison between the experimental and regresses vertical displacements ( $v$ ) near the crack tip.

Fig. 4, shows the evolution of the calculated  $K_I$  as a function of the applied load during a complete loading-unloading cycle for all the investigated operating temperatures. As shown, all the curves exhibit two different slopes: a lower slope for load values lower than 50 N and an higher one for load values between 50 N and 300 N. This behavior can be attributed to the unique non-linear stress-strain behavior of NiTi alloys as well as to the crack opening and closure mechanisms.

Furthermore, it is possible to observe that the higher the operating temperature the lower the recorded stress intensity factor. In Fig. 5, the stress intensity factor, recorded at the maximum applied load ( $P=300$  N), is plotted in function of the temperature. Results revealed that  $K_I$  tends to decrease by increasing the temperature until the material exhibits pseudoelastic properties ( $T < 320$  K). For higher values of temperature ( $T > 320$  K), transformation mechanisms tends to

vanish because the energy required to create slip plane is lower than the one needed to induce phase transformation, therefore the stress intensity factor ( $K_I$ ) tends to stabilize.

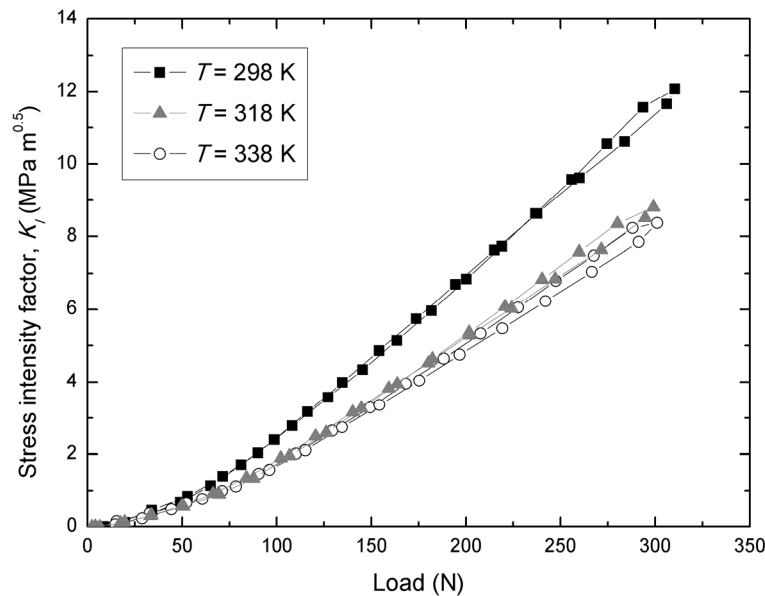


Figure 4: Evolution of the calculated Stress Intensity Factor (SIF) in a cyclic test for different operating temperatures.

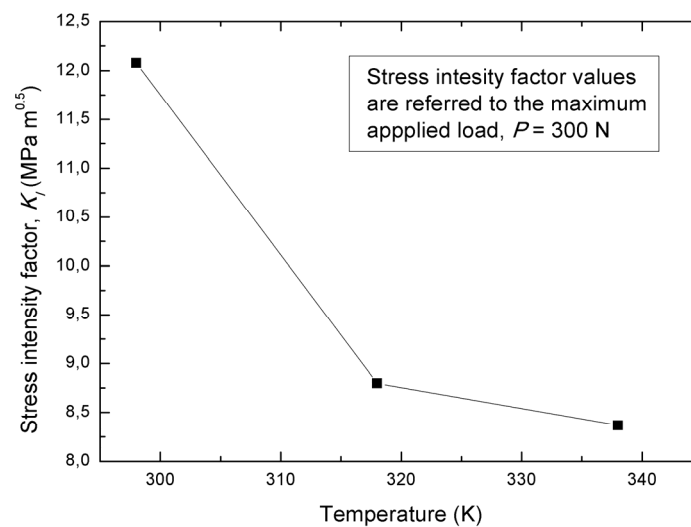


Figure 5: Evolution of the Stress Intensity Factor (SIF), recorded at the maximum applied load ( $P=300$  N), as a function of the operating temperature.

## CONCLUSIONS

Digital image correlation technique was adopted in this investigation to study the phase transition mechanisms at the crack tip in NiTi alloys. To this aim Single Edge Crack (SEC) specimens, obtained from a commercial NiTi sheets with pseudoelastic behavior, were analyzed. DIC method was used to analyze the displacement field in the crack tip region and a fitting procedures, based on the William's series expansion, were properly implemented to estimate the mode I Stress-Intensity Factor (SIF). The effect of the operating temperature on the SIF was investigated and results showed that the stress intensity factor decrease by increasing the temperature and it tends to stabilize when the pseudoelastic properties of the alloy vanish.



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